

# UNIVERSITY OF WASHINGTON

## CERAMIC ENGINEERING

COLLEGE OF ENGINEERING

SCHOOL OF MINERAL ENGINEERING

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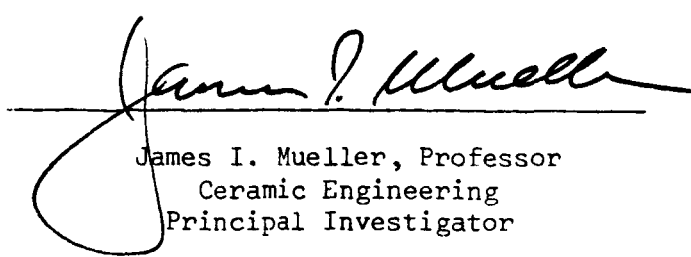
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UNIVERSITY OF WASHINGTON  
College of Engineering  
Ceramic Engineering Division

Multidisciplinary Research Activity  
in the Materials Sciences with  
Emphasis on Ceramic Materials

NASA Research Grant Number NsG-484

Semiannual Status Report Number 7  
June 16, 1966 through December 15, 1966



James I. Mueller, Professor  
Ceramic Engineering  
Principal Investigator

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## INTRODUCTION

The Ceramic Materials Research Program at the University of Washington was established June 1, 1963, under National Aeronautics and Space Administration Grant Number NsG-484. The principal purposes of the grant are to encourage multidisciplinary research upon the nature and properties of ceramic materials and to assist this institution in the development of an enduring research capability in ceramics and ceramic engineering. The funds are, therefore, used to make financial support available for research on ceramic materials conducted by members of the University faculty and to purchase such items of capital equipment as are deemed desirable for the implementation of the stated purposes.

The program, planned to study the effects of various energy environments upon ceramic materials, is divided into several broad research areas, namely; chemical, surface phenomena, solid state ceramics, processing, and radiation effects. The research program is planned and coordinated by the Ceramic Materials Research Committee, the membership of which is appointed by the Dean of the Graduate School. The current membership includes James I. Mueller, Ceramic Engineering, Chairman and Principal Investigator; Norman W. Gregory, Chemistry; John L. Bjorkstam, Electrical Engineering; O. J. Whittemore, Jr., Ceramic Engineering; and Barry D. Lichter, Metallurgical Engineering. Administration of the program is coordinated by a board consisting of E. C. Lingafelter, Associate Dean of the Graduate School, Chairman; D. E. McFeron, Chairman, Committee on Research Policy, College of Engineering; D. A. Pifer, Director of the School of Mineral Engineering and James I. Mueller, Principal Investigator.

GENERAL PROGRAM REPORT

This is the seventh semiannual status report and covers the first half of the third year of operation under this grant. During the report period, a total of twenty-nine projects were supervised by fourteen faculty members in six academic disciplines of the University. A tabulation relating academic departments to research areas and to the number of individuals receiving support will be found in Appendix A.

The Ceramic Materials Research Seminar, a period devoted to discussions of concepts and research of interest to the program, met for a total of eleven sessions during the past six months. The speakers included six graduate students, two University faculty members and three visitors. The latter included Mr. Ralph Barnett, Illinois Institute of Technology, Professor J. P. Roberts, University of Leeds, and Dr. Kiro Zmbov, Rice University.

Funds from the grant were used to supplement University support of a visiting professor during Summer Quarter. Dr. Robert G. Lye, RIAS Institute of Advanced Studies, Martin Co., Baltimore, Maryland, offered a four-week, three credit course during the "b" term of Summer Quarter entitled "Electronic Band Structure of Solids." A total of nineteen from four different departments were enrolled. In addition, Dr. Lye spent many hours in private and group discussions with faculty members and graduate students considering problems of mutual interest in their research problems. One tangible result of Dr. Lye's visit is increased interest in solid state aspects of the total research program. This is evidenced by some of the current project reports and additional indications will be observed in future aspects of the overall program.

Dr. James D. Siegwarth was added to the Ceramic Engineering staff as Senior Research Associate during the report period. Dr. Siegwarth received his doctorate in Physics from the University of Washington in June 1966, having been supported by grant funds for his research on Mossbauer Studies of nickel oxide. He is currently assisting on the photolysis study and planning coordinated research with several other faculty members on solid state ceramic problems. A new member of the metallurgical engineering faculty, Dr. Thomas G. Stoebe, has joined the program, being supported for solid state research upon defect properties of ionic crystals.

Several items of capital equipment were received during the past six months. These include an electronic circuit panel for the high temperature diffraction unit; two Leco gas analyzers for the determination of carbon and oxygen in solids; a quartz spring microbalance and high temperature equilibration furnace for gas-solid studies; two Cahn electrobalances, one for oxidation studies and one for the high temperature calorimeter; and a Spex 1500 ultra violet spectrometer. Some of these items are already in operation while others are in various stages of installation. Additional components have been received for equipment being constructed locally which are described in the individual status reports.

The program supported the attendance of twenty-four faculty members to a total of eight technical meetings of which thirteen papers were presented based

upon work supported by the grant. Thesis and papers published or presented resulting from work supported wholly or in part by the grant are listed with the individual status reports and in Appendices C and D.

In an attempt to develop better communication between personnel in this program and individuals having similar research interests outside the University, a number of the latter were invited to attend the annual autumn program review. In addition to the technical monitor from NASA, a total of twenty attended a briefing by the research supervisors and visited the various research laboratories to exchange information with our research personnel. A more detailed report is included as Appendix E.

One of the purposes of this grant was to assist in increasing the number of graduate students working towards the doctorate in ceramics or ceramic engineering. Since the program is midway through the fourth year, a report of its effect upon these graduate enrollments seems to be in order. The first individual to complete his requirements for the Ph.D. in ceramic engineering at this institution did so during the report period. Whereas only two had entered the doctorate program during the first half year under the grant, there are currently ten enrolled at the present time. A graph showing the year by year increases is shown in Appendix F.

RESEARCH STAFF

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V. V. Stringer, Secretary  
T. W. Woller, Machinist

## STUDIES OF THE ZIRCONIUM-OXYGEN-CARBON SYSTEM

This system has been selected as the subject of a general study of the effects of the chemical environment upon ceramic materials.) Several faculty members from various disciplines are participating in a coordinated study to obtain basic knowledge regarding this system.

### GAS-SOLID EQUILIBRIUM

James I. Mueller  
Professor, Ceramic Engineering

The composition and pressure of the gaseous phase(s) associated with the solid phases at various temperatures materially affects the equilibrium of a system. It is the purpose of this research to study the effects of these variables upon the Zr-O-C system.

### Studies of the Zirconium Dioxide-Carbon Reaction

S. K. Sarkar  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

Previous work has indicated the evidence of a condensed phase, tentatively identified as  $\text{ZrO}_{0.5}\text{C}_{0.5}$ . The purpose of the continuing study is to obtain additional information upon the nature of the reaction involved.

The high temperature x-ray furnace and associated vacuum equipment was moved to another laboratory and installed with a separate diffractometer and circuit panel. This will eliminate the necessity of dismantling equipment due to time sharing in another x-ray unit. Tantalum resistor-sample holders are being carburized to reduce side reactions between the principal reactants and the tantalum.

In addition to reaction studies, the graphite tube furnace will be used to produce additional material in order that additional characterization may be made.

A paper entitled "A Study of the Ternary Zr-O-C System" was presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

### Thermochemical Study of Zirconium Oxy-Carbide

A. D. Miller

Predoctoral Associate, Ceramic Engineering (June 16 to December 15)

Ph.D. Thesis Research

The objective of this study is the determination of the existence and properties of a proposed ternary compound of approximate composition  $\text{ZrC}_{0.5}\text{O}_{0.5}$  in the system Zr-C-O. The plan is to prepare very pure zirconium carbide and equilibrate it with gaseous atmospheres of small but known oxygen partial pressures at high temperature. This objective was extended to include studies of the electronic band structure of zirconium-oxygen-carbon phases. While qualitative, these should be valuable for gaining an insight as to the nature of chemical bonding existing in the compounds.

A qualitatively correct band structure was calculated for ZrC by the tight-binding method. This structure will be modified by performing the same computation but substituting oxygen-like wave functions for a fraction of the carbon functions. A number of such calculations will be made to determine the effect of increasing oxygen content on the band structure and density-of-states.

The experimental work on equilibration of ZrC with oxygen containing atmospheres is in the data-taking phase. The equipment was assembled and calibrated during this period. A method of preparing zirconium carbide was developed in which a zirconium wire is directly carburized by contact with pyrolytic graphite deposited on its surface by the decomposition of methane.

### Influence of Oxygen Activity on the Structure of Zirconium Oxide

K. M. Nair

Predoctoral Associate, Ceramic Engineering (June 16 to December 15)

Ph.D. Thesis Research

The purpose of this study is the determination of the effects of low oxygen partial pressure at high temperature upon the stability of  $\text{ZrO}_2$ . It is planned to study the possible existence of lower oxides of zirconium, their dependence upon oxygen activity and temperature, and the influence of the formation of such an oxide upon the formation of a ternary compound in the Zr-C-O system.

The results of x-ray analysis of reaction products obtained to date are inconclusive. Some consistent diffraction lines have been observed but they are not related to the published patterns of  $\text{ZrO}_2$ , ZrC, ZrN or  $\text{ZrO}_{0.5}\text{C}_{0.5}$ . The high temperature, high vacuum thermal gravimetric unit is being modified for use in this study.

### Diffusion Study of a $\text{ZrO}_2$ -C Couple

Charles A. Petschke

Research Assistant, Ceramic Engineering (June 16 to August 10)

M.S. Thesis Research

This investigation was initiated to study the characteristics of a zirconium dioxide-carbon diffusion couple. It is hoped that the results may lead to information which will aid in understanding reactions in the Zr-C-O system.

Polished discs of pure graphite and pure monoclinic  $\text{ZrO}_2$  were hot pressed in an argon atmosphere at 1600°C, 1700°C, 1800°C and 1900°C to form diffusion couples. These were kept at temperature for a period of five hours after which the couples were sectioned for examination with a microscope, x-ray diffractor and electron microprobe. No diffusion of zirconium into the graphite was observed and no diffusion of carbon into zirconium dioxide was noted at the lower three temperatures. At 1900°C the carbon diffused into the zirconia and reacted to form a condensed phase consisting of zirconium oxygen and carbon. A ten-hour hold at 1900°C produced an interface of this material whose thickness was about four times that obtained with a five-hour hold. The diffraction peaks of the interface material closely matched the lines obtained for the ternary compound  $\text{ZrO}_{0.5}\text{C}_{0.5}$ .

Charles A. Petschke received an M.S. in Ceramic Engineering, August 1966. Thesis title: "A Study of a Zirconium Dioxide-Carbon Diffusion Couple."

### Oxygen Solubility in Zirconium Carbide

H. Peter Beaupain (Unsupported)

Part-time Graduate Student, Ceramic Engineering

M.S. Thesis Research

The purpose of this research is to determine the extent of oxygen solubility in zirconium carbide under varying conditions of temperature and oxygen pressure. This work was initiated under grant support but has been inactive until recent months.

Controlled atmosphere at high temperature will be utilized to obtain ZrC-O solid solutions. The amounts of solubility and their effects will be studied by means of x-ray diffraction, electron microprobe and thermal gravimetric techniques.

## SOLID-SOLID EQUILIBRIUM

N. W. Gregory  
Professor, Department of Chemistry

A thermodynamic and kinetic study of chemical reactions in oxide-carbide-graphite systems.

Torsion Effusion Studies of Oxide-Carbide-Graphite Systems

Juey Hong Rai  
Research Assistant, Chemistry (June 16 to December 15)  
Ph.D. Thesis Research

Mr. Rai has just begun experimental work during the past six months. He will use equipment assembled by previous workers on this project. He has been calibrating the torsion effusion apparatus as well as familiarizing himself with its operation by measuring the vapor pressure of copper, silver and gold, respectively. He has begun some preliminary studies on the interaction of MgO and graphite.

A paper entitled "A Torsion Effusion Study of the Reaction of Graphite with Hafnium and Uranium Dioxides," by T. C. M. Pillay and N. W. Gregory was published in the Journal of Physical Chemistry, 70, 3140 (1966).

## CALORIMETRIC INVESTIGATION OF CERAMIC AND RELATED MATERIALS

Barry D. Lichter  
Associate Professor, Metallurgical Engineering

The objectives are the development of a high temperature calorimetric facility for the study of heat capacities and heats of transformation of ceramic and related materials.

High Temperature Drop Calorimetry

Hugo W. Schimmelbusch  
Research Assistant, Metallurgical Engineering (September 16 to December 15)  
M.S. Thesis Research

The object of this program is the design and construction of a high-temperature diphenyl-ether drop calorimeter. The following calorimeter components and systems are required: (1) calorimeter receiving vessel, (2) room temperature thermostat bath, (3) calorimeter gates, (4) sample drop

mechanism, (5) platinum-rhodium resistance furnace, (6) calorimeter supporting members and counterweight system, (7) power supply and furnace temperature control, (8) microbalance for mercury displacement measurement, and (9) vacuum inert gas train system.

In Status Reports Nos. 5 and 6 the final design and the construction or procurement of items 1 and 2 have been reported, as well as the final design of items 3 through 6. During the current report period, items 7 and 8 have been procured and initial design of item 9 has been carried out. The microbalance (item 8) has sufficient sensitivity to be used during the "rating" periods before and after sample drop as well as suitable capacity to record the maximum total mercury displacement. In addition two water-cooled, vacuum operating gates have been constructed, all components for furnace construction have been obtained, and a novel magnetic-brake has been designed for the drop mechanism. Final purification of diphenyl-ether (fractional crystallization) has been completed, and a system for adding the mercury and ether to the calorimeter under vacuum has been designed.

An invited paper entitled, "Review of High Temperature Calorimetric Techniques" was presented by B. D. Lichter at the Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

#### THERMODYNAMICS OF TERNARY CERAMIC PHASE DIAGRAMS

Gerald W. Toop  
Assistant Professor, Metallurgical Engineering

The object of this investigation is to attempt to predict phase boundaries in ternary phase diagrams using the thermodynamic properties of the binary systems.

#### Ternary Ceramic Phase Diagrams

G. Harinarayanan  
Research Assistant, Metallurgical Engineering (June 16 to October 31)  
M.S. Thesis Research

Dong Nyung Lee  
Research Assistant, Metallurgical Engineering (September 16 to December 15)  
M.S. Thesis Research

This work is continuing by applying the method of calculation to ternary systems involving refractory metal oxides and carbides for which binary thermodynamic data is available. The calculation technique has been improved considerably by the development of graphical solutions to the equations. The method has been shown to give good results for condensed ternary systems

exhibiting immiscibility gaps or a tendency toward compound formation. The method is least accurate for ternary systems with highly stable, distinct, ternary compounds.

G. Harinarayanan received an M.S. in Metallurgical Engineering, December 1966. Thesis title: "Prediction of Isothermal Phase Boundaries in Ternary Phase Diagrams using Binary Thermodynamic Data."

A paper entitled "Predicting Ternary Phase Diagrams Using Binary Data" was presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

## ZIRCONIUM OXIDATION

Thomas F. Archbold  
Assistant Professor, Metallurgical Engineering

The objectives of this project are to determine the nucleation and growth characteristics of oxide(s) on zirconium metal at elevated temperatures, as well as to identify and characterize the oxide(s) by electron diffraction. Short-time heating by radiation will take place at reduced oxygen pressures.

### Zirconium Oxidation

L. P. Srivastava  
Predoctoral Associate, Metallurgical Engineering (June 16 to December 15)  
Ph.D. Thesis Research

The details of the initial stages of the oxidation of zirconium are being investigated. The gas purification system has been completed and tested. A considerable amount of effort was devoted to determine the proper experimental conditions with regard to the Cahn Electrobalance. Convection currents near the specimen surface during short-time heating has been a difficult problem as it creates unsatisfactory noise levels in the balance. Modifications in the method of heating are being considered in order to minimize the noise.

Techniques for the replication of oxide-free and lightly-oxidized metal surfaces have been perfected. The oxide stripping technique for relatively thick oxide layers has been modified to yield undisturbed oxide films for examination in the electron microscope. Preliminary results indicate that all of the oxides are crystalline, in contrast with results reported by other workers. Also, the electron diffraction patterns appear to be different than those appearing in the literature.

An attempt is being made to determine oxide thicknesses directly on the microscope in order to aid the evaluation of the effects of defect structures on the kinetics of oxidation.

## SOLID STATE CERAMICS

The bulk properties of some ceramic materials are of prime importance and these are, for the most part, dependent upon structural considerations. The purpose of this research is to relate the nature of crystalline ceramics with appropriate properties.

### FERROELECTRIC MATERIALS STUDY

Robert J. Campbell, Jr.  
Assistant Professor, Ceramic Engineering

The objective of these studies is the development of an explanation of the mechanism of energy storage in ferroelectric materials, resulting from displacement of atoms in the crystal lattice.

### Effect of Foreign Atoms in the Perovskite Lattice on Electrical Properties

Fennimore N. Bradley  
Predoctoral Associate, Ceramic Engineering  
Ph.D. Thesis Research

The objective of this study is to determine the effect of ionic impurities upon the energy storage behavior of ferroelectrics and, in particular, to determine the effect of impurities upon the Curie temperature of perovskite ceramics and to establish the mechanism of this phenomenon.

As previously determined this study has shown that the differing properties ascribed to various dopants is almost entirely due to their effect on the grain size of three different ferroelectric titanates. A mechanism which relates internal strain produced within extremely small grains to dielectric properties was demonstrated. The release of this strain by grinding the fired fine-grain material restored tetragonality to the crystals.

F. N. Bradley received the Ph.D. in Ceramic Engineering, December 1966.  
Dissertation title: "Ion Doped Perovskite Ceramics."

### Time Dependent Properties

Tracy L. Scott  
Research Associate, Ceramic Engineering (June 20 to December 15)  
M.S. Thesis Research

Studies of changes of dielectric constant with time were related to grain size of  $\text{BaTiO}_3$  samples produced at the same firing temperatures. Grain size was controlled by additions of iron as indicated by the work of Bradley.

Studies to date indicate that the aging rate varies inversely with grain size. The reason for this departure from the results of other investigators has not been determined.

Samples for this study have been dry pressed but ultimate density of the specimens was very low (60-70% of theoretical). Dies and punches for hot pressing are being acquired so that better control of grain size and final density may be accomplished.

#### Differentiation of Surface and Grain Boundary Effects

Robert J. Mauzy

Research Associate, Ceramic Engineering (September 16 to December 2)

M.S. Thesis Research

This study is intended to develop an explanation of the differences observed in properties of ferroelectrics crystals containing impurities. The knowledge of the location of the impurity atom, whether within the crystal or contained in the grain boundaries, should provide an immediate answer to the role of these impurities.

Studies of polycrystalline ferroelectrics containing known additions of impurities by the electron microprobe are expected to assist in locating the atoms. A different phase of this study would provide single crystals with impurities diffused into the structure for measurement of intra-crystal effects caused by the impurities.

Mr. Mauzy has found it necessary to interrupt his research for at least one year for personal reasons. His studies to date were of library colation and have not revealed any new problems.

#### Ferroelectric Structure

Richard E. Deno (Office of Engineering Research)

Research Assistant, Ceramic Engineering

M.S. Thesis Research

An attempt is being made to correlate structural effects on polarization by subjecting ferroelectric crystals to mechanical deformation during polarization. It is hoped that this research may lead to a better understanding of the mechanical-electrical interrelation.

### Oxidation Studies in Colored Glasses

James W. Vogan (Unsupported)  
Part-time Graduate Student, Ceramic Engineering  
M.S. Thesis Research

A study of electrochemical potentials is expected to reveal the influence of oxidizers and firing atmospheres on the color of amber glasses. It is also hoped that a better explanation of the role of sulfur and carbon in these glasses may be developed.

### DOMAIN DYNAMICS IN ISOMORPHOUS FERROELECTRICS

John L. Bjorkstam  
Professor, Electrical Engineering

The objective of this investigation is to study the effect of deuterium substitution on domain properties of hydrogen bonded ferroelectrics, and to correlate the results with a microscopic model for the domain wall and the process of domain reversal (switching).

### Ferroelectric Domains and Domain Motion in $\text{KH}_2\text{PO}_4$ and $\text{KD}_2\text{PO}_4$

Richard E. Oettel  
Predoctoral Associate, Electrical Engineering (June 16 to December 15)  
Ph.D. Thesis Research

The improved apparatus mentioned in the previous semiannual report is working very well and data acquisition is proceeding normally. Our primary measurement is the sidewise velocity of a  $180^\circ$  domain in a region of opposite polarization. Domain velocities are being obtained as a function of temperature and degree of deuteration. One rather interesting feature already apparent is the large increase in domain mobility with a small departure from complete deuteration. For example, a crystal which is 90% deuterated has a mobility only about one order of magnitude less than the completely undeuterated crystal, while there is an approximately six-order of magnitude decrease in mobility from pure  $\text{KH}_2\text{PO}_4$  to pure  $\text{KD}_2\text{PO}_4$ . The very much greater rate at which hydrogens can reorient within the unit cell during polarization reversal is responsible for this profound isotope effect. A very small fraction of hydrogens in the crystal can, so to speak, short out the sluggish deuterium reorientation mechanism and provide the dominant path through which domain growth progresses.

We have shown that an atomic model for the wall of a long needle shaped  $180^\circ$  domain, such as we observe, must have a different arrangement of atoms in the side walls than in the tip. The electrostatic energy per unit area of the tip wall is  $\sim 50\%$  greater than for the side wall. We believe this to be one reason why domains grow by extending the side wall rather than the tip wall.

A paper entitled "The Effect of Deuteron Substitution on Domain Dynamics in Potassium Dihydrogen Phosphate," by John L. Bjorkstam and Richard E. Oettel was presented at the International Meeting on Ferroelectricity, Prague, Czechoslovakia, June 28-July 1, 1966.

A paper entitled "Domain Mobility in Hydrogen-Bonded Ferroelectrics," by Richard E. Oettel and John L. Bjorkstam was presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

#### PHASE TOPOGRAPHY

Jerry E. Turnbaugh  
Assistant Professor, Ceramic Engineering

Selected microstructural features common to ceramics are being studied in terms of the variables leading to their development.

#### Phase Topography

Pei Sung  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

All experimental apparatus is now functioning and has been calibrated. The CEC 24-120B Leak Detector has been successfully adopted to measurements of the  $\text{He}^3/\text{He}^4$  ratio. Preliminary solubility measurements of  $\text{He}^4$  in  $\text{UO}_2$  give results which appear to agree with literature values. Measurements of saturation solubility of  $\text{He}^4$  in  $\text{UO}_{2.01}$  are now being made on 10- $\mu\text{m}$  particles at 1500°C and 100-atm.

#### Volume Stress Research

Experimental work has been terminated with the completion of Mr. Anderson's thesis requirements. A more elaborate version of Buessem's model for internal stresses in polycrystalline materials has been developed which takes stress relaxation by creep into account. The model has yet to be proven.

A paper entitled "Thermal Expansion of Beta-Eucryptite" by Norman C. Anderson and Jerry E. Turnbaugh was presented at the Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

## MOSSBAUER STUDIES

J. G. Dash  
Professor, Department of Physics

Relations between lattice dynamics and electronic configurations  
in  $\text{FeF}_2$  and  $\text{FeCl}_2$ , by means of the Mossbauer effect.

### Solid State Studies

Duane P. Johnson  
Predoctoral Research Associate, Department of Physics (June 16 to December 15)  
Ph.D. Thesis Research

This study is part of a continuing experimental program of application of the Mossbauer effect to solid state studies.

Measurements of the Mossbauer fraction, quadrupole splitting, and isomer shift of  $\text{Fe}^{57}$  in thin absorbers of  $\text{FeF}_2$  and  $\text{FeCl}_2$  have been made at temperatures between  $4^\circ$  and  $300^\circ\text{K}$ . This has required the development of techniques for producing thin, uniform powdered samples of known thickness. Methods have also been developed for measuring splittings and shifts to appreciably higher accuracy than quoted in published studies. The measurements on  $\text{FeCl}_2$  appear to be complete:  $\text{FeF}_2$  will be completed shortly. Theoretical analysis, in terms of crystal field theory, is in an early, but promising stage. It is hoped, but not yet certain, that we will be able to describe the atomic force constants in terms of the atomic configurations of the Fe atoms.

A paper entitled "The Mossbauer Effect in  $\text{Fe}^{57}$  in  $\text{MnO}$  and  $\text{NiO}$ " by J. D. Siegwarth was presented at the American Physical Society, Minneapolis, Minnesota, June 20-22, 1966.

A paper entitled "The Mossbauer Effect of Divalent  $\text{Fe}^{57}$  in  $\text{NiO}$  and  $\text{MnO}$ " by J. D. Siegwarth has been accepted for publication by The Physical Review.

## DEFECT PROPERTIES

Thomas G. Stoebe  
Assistant Professor, Metallurgical Engineering

This study is designed to study the influence of lattice defects on the properties of  $\text{LiF}$  and  $\text{MgO}$  crystals, with particular attention to the growth of high purity single crystals.

## Defect Properties of Ionic and Ceramic Crystals

Hira L. Fotedar (Office of Engineering Research)  
Graduate Assistant, Metallurgical Engineering  
M.S. Thesis Research

Accomplishments during this report period include preparation of existing equipment and the ordering of temperature control apparatus for crystal growth. Literature surveys are being conducted concerning purification and analysis methods for LiF, methods of growth of MgO and mechanical properties measurements in LiF.

### MECHANISMS OF DEFORMATION

Donald J. Bailey  
Senior Research Associate, Metallurgical Engineering

The dynamical theory of plastic deformation, which phenomena logically accounts for the multiplication and stress-dependent velocity of dislocations, has been shown to accurately reflect the mechanistic behavior of deformation. The detailed behavior of the empirical parameters, and the influence of deformation substructural changes, has been predicted from theory and agrees well with experiment. The parameter relating dislocation velocity to the stress has been shown to be independent of the state of deformation provided it is properly interpreted in terms of the effective stress acting on dislocations; the specific relationship of this effective stress to the mobile dislocation density is found to be uniquely characteristic of the rate controlling deformation mechanism. Detailed consideration of the relationships of the dynamical theory, together with the influence of impurities on the deformation process led to a model which satisfactorily predicts deformation behavior in a dynamically strain-aging material.

A paper entitled "The Relationship Between Dislocation Density and Flow Stress in Materials Deforming by a P.-N. Mechanism" by W. F. Flanagan and D. J. Bailey has been accepted for publication in the Philosophical Magazine.

A paper entitled "X-ray Analysis of Fatigue in Copper" by W. F. Flanagan, R. Baggerly and R. Pelloux has been accepted for publication in Advances in X-ray Analysis, 10 (Plenum Press).

A paper entitled "X-ray Analysis of Fatigue in Copper" by W. F. Flanagan, R. Baggerly and R. Pelloux was presented at the 15th Conference on X-ray Analysis at Denver Colorado, August 10-12, 1966.

## SURFACE PHENOMENA

Many properties of ceramic materials are governed by the character of the grain boundaries of the polycrystalline composite. This research area was established to obtain fundamental information regarding the chemistry and physics of the surfaces of ceramic materials.

### ALUMINUM OXIDE BICRYSTALS

William D. Scott  
Assistant Professor, Ceramic Engineering

The purpose of this research is to study grain boundary effects in macroscopic bicrystals of aluminum oxide by controlling misorientation and environment during their preparation.

### Mechanical Properties of Aluminum Oxide Bicrystals

Henry Y. B. Mar  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

The purpose of this project is to produce aluminum oxide bicrystals with controlled misorientation and to study the mechanical properties of the grain boundaries as a function of misorientation, stress and temperature.

A molybdenum and graphite die system has been developed to obtain axial alignment in the hot-press during bicrystal fabrication, and alumina bicrystals have been successfully fabricated at 2000 p.s.i. at 1400°C for two hours. Work is continuing on the evaluation of the quality of the boundaries produced.

We are attempting to fabricate aluminum oxide bicrystals by pressure-sintering in clean vacuum conditions to produce clean grain boundaries with closely controlled misorientation. These specimens will then be used for mechanical properties tests as well as in the investigations on pore removal and interfacial energy.

### Pore Growth and Pore Removal in Grain Boundaries of Aluminum Oxide

William D. Scott  
Assistant Professor, Ceramic Engineering

Chris T. McLeod  
Undergraduate Aide, Ceramic Engineering (June 16 to December 15)

The purpose of this project is to study the formation and subsequent removal of pores located on grain boundaries in aluminum oxide bicrystals, and to relate this pore behavior to sintering and diffusion in aluminum oxide.

A method has been found to separate sections of bicrystals along the boundary after extended heat treatment. Pores located on the boundary were found to be highly polygonized, and low energy planes forming the pore surfaces have been identified by combined x-ray diffraction and optical and electron microscopy techniques.

#### Interfacial Energies of Aluminum Oxide Bicrystals

James F. Shackelford  
Research Assistant, Ceramic Engineering (September 16 to December 15)  
M.S. Thesis Research

The purpose of this project is to determine the relative interfacial grain boundary energies in aluminum oxide by measuring the profile of thermally etched boundaries.

A simple tilt boundary with misorientation selected to produce pure edge dislocations will be investigated and results will be compared with current theories on grain boundary structures.

#### The Effect of MgO Impurity on Relative Interfacial Energy in Alumina

Michael Matson (Unsupported)  
Part-time Graduate Student, Ceramic Engineering  
M.S. Thesis Research

The purpose of this project is to measure the effect of the presence of MgO on the grain boundary energy of alumina and to correlate this effect with current theories of sintering in alumina.

Several direct methods of measuring the profile of a thermally etched boundary have been investigated. Electron microscopy and surface profile measurements were found to lack the required sensitivity. Interferometric techniques will be used. A high purity, single crystal of alumina has been obtained, and MgO doped material is on order.

#### Impurity Diffusion in MgO Under the Influence of an Electric Field

Chester A. Hinman  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

The purpose of this project is to investigate the diffusion of nickel in MgO at high temperature and in an electric field. By using the microprobe to measure the drift of the diffusion profile in a sandwich diffusion couple, one can obtain, with the help of other bulk diffusion data, the mobility and effective charge of the nickel ions. Information can also be obtained on the diffusion mechanism.

A Centorr furnace for use at 2000°C in controlled oxygen pressures is on order, and alumina and platinum components for the specimen holder have been received. Single crystal MgO specimens with matched, cleaved surfaces have been received. Initial microprobe measurements on diffusion profiles in polycrystalline MgO are being made to develop quantitative analysis techniques.

#### SURFACE DIFFUSION

Jerry E. Turnbaugh  
Assistant Professor, Ceramic Engineering

James M. Isaacson,  
Undergraduate Aide, Ceramic Engineering (June 16 to August 31)

Impurity surface diffusion coefficients are to be measured in order to obtain information upon the structure and chemical nature of the surfaces of ceramic materials.

Construction of apparatus is continuing. No experimental work has been accomplished.

#### USE OF CERAMICS IN CONTROLLING ANTENNA SYSTEM PARAMETERS

Irene C. Peden  
Associate Professor, Electrical Engineering

H. Myron Swarm  
Professor, Electrical Engineering

The purpose of this research is to study the characteristics of ceramics as modelling materials for antenna systems.

### Ceramic Models of the Lunar Surface

Monte R. Mueller

Undergraduate Aide, Electrical Engineering (June 16 to August 31)

Information as to the composition of the lunar surface might be gained by analyzing radio signals transmitted from the moons surface in terms of the electrical properties of the surface material.

The purpose of this work was to make initial determinations of the relative dielectric constant and loss tangent of dunite (olivine) over a frequency range of 2 to 12 GHz. It was desired to establish possible relationships between relative dielectric constant and loss tangent versus frequency and density.

Preliminary studies indicate that it may be impractical to use a balanced transmission technique to resolve the relative dielectric constant at any frequencies higher than X band for samples more granular than a fine powder.

The project is currently inactive due to the lack of a graduate student to continue this work.

### Ceramic Model of the Earth Ionosphere Waveguide System

George Webber

Research Assistant, Electrical Engineering (June 16 to September 15)

M.S. Thesis Research

The objective is to study the influence of ionospheric electron density perturbations on very low frequency propagation by use of various ceramic materials in models operating at microwave frequencies.

The general theory and model have been completed.

George Webber received his M.S. in Electrical Engineering. Thesis title: "A Mode Analysis of Very Low Frequency Radio Wave Propagation Using Ceramic Dielectric Models of the Earth-Ionosphere Waveguide."

### Characterization of Dielectric Materials

O. J. Whittemore, Jr.

Associate Professor, Ceramic Engineering

The objective of this study is to characterize the chemical and physical properties of dielectric ceramics, particularly those of interest in the antenna models studied by Peden and Swarm.

S. P. Banerjee

Undergraduate Aide, Ceramic Engineering (September 15 to December 15)

A report entitled "Characterization of Composite Dielectrics" was prepared, which correlates physical properties of carbon:silica:air mixtures with dielectric properties.

## EFFECTS OF RADIATION UPON CERAMIC MATERIALS

Electromagnetic spectral radiation causes varying physical and chemical effects upon materials. The nature of the effects is usually a function of the energy of the incident radiation and the character of the material. It is the purpose of this research to more accurately characterize these properties with the nature of ceramic materials.

### ULTRA-VIOLET PHOTOLYSIS OF CERAMIC MATERIALS

James I. Mueller  
Professor, Ceramic Engineering

The purpose of this research is to relate the energy absorption with the electronic interactions in ceramic materials.

#### U-V Photolysis Study

James D. Siegwarth  
Senior Research Associate, Ceramic Engineering

Jack K. Merrow  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

Lakshmi Annapoorni  
Research Assistant, Ceramic Engineering (September 16 to December 15)  
Ph.D. Thesis Research

The initial study of ultraviolet effects on MgO will be done using optical techniques. The wave lengths of the luminescent and thermoluminescent light emitted from ultraviolet irradiated MgO will be measured using a Spex 1500 spectrometer. The UV light used for irradiation will be monochromatized by a double grating monochromator consisting of a coupled pair of Jarrell-Ash 0.25 meter Ebert monochromators.

The Spex UV spectrometer has been delivered in part and some of the required electronic components have been received also. Parts have been ordered for the liquid nitrogen temperature dewar system in which the sample will be placed for irradiation. The dewar and associated vacuum system is being designed presently.

One of the major difficulties encountered when attempting to understand optical effects in MgO has been the low purity of the material. The purest single crystals available now seem to be those supplied by Monocrystals Company of Cleveland, Ohio. Their crystals are small but the impurity level is about 0.01 atomic percent with an impurity of Fe only about 0.001 atomic percent. Three of these crystals have been ordered for our use.

PROCESSING

Research in this area is intended to gain information relative to the effect of processing variables upon the micro- and macro-structure of ceramic materials and upon their resultant properties.

## CERAMIC PROCESSING

O. J. Whittemore, Jr.  
Associate Professor, Ceramic Engineering

Initial Stages of Sintering

J. Joseph Sipe  
Research Assistant, Ceramic Engineering (June 16 to December 15)  
M.S. Thesis Research

C. Peter Becker  
Undergraduate Aide, Ceramic Engineering (June 16 to September 15)

The objective of this project is to study the initial stages of sintering where pore growth occurs. Several ceramic materials have shown this phenomenon during sintering. Auxiliary objectives are to determine if it occurs generally and mechanisms controlling it.

During the sintering of compacts of fine particles of magnesia and ferric oxide, pore growth has been shown to occur simultaneously with densification. Similar compacts of alpha alumina showed only densification during sintering. Time temperature studies are being completed on these three oxides.

Characteristics of Plasma-Sprayed Alumina

Vere S. Thompson (Unsupported)  
Part-time Graduate Student, Ceramic Engineering  
M.S. Thesis Research

Plasma-sprayed alumina is deposited as gamma alumina rather than the normal alpha form. The objective of this project was to study the effects of reheating samples of plasma-sprayed alumina.

The conversion of gamma to alpha alumina was found to occur after heating to about 1260°C and to be accompanied by a five to six-fold increase in total pore volume with only 1% linear shrinkage. Pore growth occurred through all of the temperature ranges studied, i.e. 1150 to 1590°C. By integrating pore size distribution curves, internal surface areas were calculated and shown to

decrease rapidly to 1260°C, remain rather constant to 1480°C, then to decrease further at 1590°C.

Vere S. Thompson received his M.S. in Ceramic Engineering, December 1966. Thesis title: "Characteristics of Plasma-Aprrayed Alumina."

A paper entitled "Structural Changes on Reheating Plasma-Sprayed Alumina" by Vere S. Thompson and O. J. Whittemore, Jr. has been accepted for presentation at the Annual Meeting of the American Ceramic Society, New York City, May 1967 (Refractories Division).

### Characterization and Forming

Douglas J. Calkins  
Predoctoral Associate, Ceramic Engineering (June 16 to December 15)  
Ph.D. Thesis Research

Daniel Leiser  
Research Assistant, Ceramic Engineering (June 16 to December 15)  
M.S. Thesis Research

George Deitrick  
Undergraduate Aide, Ceramic Engineering (June 16 to August 15)

The objective of this project is to study ceramic forming methods and correlated characterization of particles and agglomerates.

The compaction behavior of ceramic particles is being studied by determining stress versus compaction curves on particles of different sizes. Glass spheres and fused alumina grains have been compacted. More recovery by elastic behavior was noted with the glass spheres while the alumina grain showed more net compaction, probably partially due to greater fracture. The sensitive dynamic curves obtained with the Instron machine have a ratcheted appearance, possibly due to the fracture during compaction.

A literature survey of the state of knowledge of the compaction process has been completed.

### SINTERING OF ICE

Peter V. Hobbs  
Associate Professor, Atmospheric Sciences

The principal objective of this program was to develop a theory for the densification of a compact of ice spheres and to check the theory experimentally.

Lawrence L. Radke  
Research Assistant, Atmospheric Sciences (June 16 to December 15)  
M.S. Thesis Research

Diana Kay Wood  
Undergraduate Aide, Atmospheric Sciences (June 16 to August 31)

Accurate measurements have been made of the rate of densification of a compact of ice spheres as a function of temperature and particle size. The results are in good agreement with theoretical predictions based on a volume-diffusion model. The theory that has been developed, and the way in which it has been checked experimentally, should be of general interest to ceramic engineers.

Lawrence L. Radke received an M.S. in Atmospheric Sciences, December 1966.  
Thesis title: "The Densification of Artificial Firn."

#### EFFECTS OF LANTHANUM DOPING UPON BARIUM TITANATE

James I. Mueller  
Professor, Ceramic Engineering

Ronald P. Burley (National Lead Co. Fellowship)  
Research Fellow, Ceramic Engineering  
M.S. Thesis Research

This investigation was initiated to determine the effects of processing parameters such as type of lanthanum additive, firing temperature and furnace atmosphere upon the micro- and macro-properties of barium titanate.

A literature survey has been completed and several titanate samples have been prepared using different lanthanum chemicals as dopant source. Microstructural characteristics and identification of condensed phases are being studied.

It is planned to use two or three different lanthanum source additives and determine the microstructural effects in order to gain additional insight upon the resistivity anomaly.

## APPENDIX A-1

Distribution of Projects Within the University According to Research Areas

<u>Academic Department</u>	<u>Number of Projects</u>	<u>Zr-O-C</u>	<u>Solid State</u>	<u>Surface</u>	<u>Rad</u>	<u>Process</u>
Atmospheric Sciences	1	-	-	-	-	1
Chemistry	1	1	-	-	-	-
Ceramic Engineering	18	4	5	6	1	2
Electrical Engineering	3	-	1	2	-	-
Metallurgical Engineering	5	3	2	-	-	-
Physics	1	-	1	-	-	-
	—	—	—	—	—	—
TOTAL	29	8	9	8	1	3

## APPENDIX A-2

Number of Students and Faculty Involved in Research Supported by Grant Funds

<u>Academic Department</u>	<u>Number of Projects</u>	<u>Faculty</u>	<u>Research Faculty</u>	<u>Under Grads</u>	<u>MS</u>	<u>Ph.D.</u>	<u>Total Grads</u>
Atmospheric Sciences	1	1	-	1	1	-	1
Chemistry	1	1	-	-	-	1	1
Ceramic Engineering	18	4	2	5	6	10	16
Electrical Engineering	3	3	-	1	1	1	2
Metallurgical Engineering	5	4	1	-	3	1	4
Physics	1	1	-	-	-	1	1
	—	—	—	—	—	—	—
TOTAL	29	14	3	7	11	14	25

## APPENDIX B

## Ceramic Materials Research Seminars

"Design with Brittle Materials"

Mr. Ralph Barnett, Illinois Institute of Technology Research Institute

"On the Relationship Between Dislocation Density and Flow Stress"

Mr. Donald J. Bailey, Senior Research Associate, Metallurgical Engineering

"Diffusion Study of a  $ZrO_2$ -C Couple"

Mr. Charles A. Petschke, Research Assistant, Ceramic Engineering

"Ion-Doped Perovskite Ceramics"

Fennimore N. Bradley, Predoctoral Associate, Ceramic Engineering

"Physical Ceramic Research at Leeds"

Dr. J. P. Roberts, Chairman of the Department of Ceramic Engineering,  
University of Leeds, Leeds, England

"Mass Spectrometry"

Professor Kiro Zmbov, Department of Chemistry, Rice University, Houston, Texas

"Sintering of Ice"

Mr. Lawrence F. Radke, Research Assistant, Department of Atmospheric Sciences

"Ionic Conductivity and Diffusion in LiF Crystals"

Dr. Thomas G. Stoebe, Assistant Professor of Metallurgical Engineering

"Observations of European Scientific Meetings"

Dr. John L. Bjorkstam, Professor of Electrical Engineering

"Pore Growth-Initial Stages of Sintering"

Mr. John J. Sipe, Research Assistant, Ceramic Engineering

"Aging of Barium Titanate Ceramics"

Mr. Tracy L. Scott, Research Assistant, Ceramic Engineering

## APPENDIX C

## Theses Published

"Ion Doped Perovskite Ceramics"

(Fennimore N. Bradley) Ph.D., Ceramic Engineering

"Prediction of Isothermal Phase Boundaries in Ternary Phase Diagrams using Binary Thermodynamic Data"

(G. Harinarayanan) M.S., Metallurgical Engineering

"A Study of Zirconium Dioxide-Carbon Diffusion Couple"

(Charles A. Petschke) M.S., Ceramic Engineering

"The Densification of Artificial Firn"

(Lawrence L. Radke) M.S., Atmospheric Sciences

"Propagation Using Ceramic Dielectric Models of the Earth-Ionosphere Waveguide"

(George E. Webber) M.S., Electrical Engineering

## APPENDIX D

## Papers Published:

"A Torsion Effusion Study of the Reaction of Graphite with Hafnium and Uranium Dioxides," T. C. M. Pillay and N. W. Gregory, Journal of Physical Chemistry, 70, 3140 (1966).

## Papers Accepted for Publication:

"The Relationship Between Dislocation Density and Flow Stress in Materials Deforming by a P.-N. Mechanism," W. F. Flanagan and D. J. Bailey, Philosophical Magazine.

"X-ray Analysis of Fatigue in Copper," W. F. Flanagan, R. Baggerly and R. Pelloux, Advances in X-ray Analysis, 10 (Plenum Press).

"The Mossbauer Effect of Divalent Fe<sup>57</sup> in NiO and MnO," J. D. Siegwarth, The Physical Review.

## Papers Presented:

"Domain Mobility in Hydrogen-Bonded Ferroelectrics," John L. Bjorkstam and Richard E. Oettel, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"The Effect of Deuteron Substitution on Domain Dynamics in Potassium Dihydrogen Phosphate," John L. Bjorkstam and Richard E. Oettel, International Meeting on Ferroelectricity, Prague, Czechoslovakia, June 28-July 1, 1966.

"Electrical Properties of Non-Stoichiometric Barium Titanate," Robert J. Campbell, Jr. and Robert E. Schultz, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"X-ray Analysis of Fatigue in Copper," W. F. Flanagan, R. Baggerly and R. Pelloux, 15th Conference on X-ray Analysis, Denver, Colorado, August 10-12, 1966.

"Review of High Temperature Calorimetric Techniques," B. D. Lichter, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"Microstructure Determinations in BeO," J. I. Mueller and Robert Thorsen, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"Monoclinic-Tetragonal Inversion of Zirconium Dioxide," J. I. Mueller and John A. Negrych, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"A Study of the Ternary Zr-O-C System," J. I. Mueller and S. K. Sarkar, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966

"Influence of Grain Size and Poling on Aging of Barium Titanate," Tracy L. Scott, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"The Mossbauer Effect in Fe<sup>57</sup> in MnO and NiO," J. D. Siegwarth, American Physical Society, Minneapolis, Minnesota, June 20-22, 1966.

"Predicting Ternary Phase Diagrams Using Binary Data," G. W. Toop, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"Thermal Expansion of Beta-Eucryptite," Jerry E. Turnbaugh and Norman C. Anderson, Pacific Coast Regional Meeting of the American Ceramic Society, Portland, Oregon, October 26-28, 1966.

"Survey of Ceramic Processing Instruction in Current Ceramic Engineering Curricula," O. J. Whittemore, Jr., American Society for Engineering Education, Pullman, Washington, June 21-24, 1966.

#### Papers Accepted for Presentation:

"Structural Changes on Reheating Plasma-Sprayed Alumina," O. J. Whittemore, Jr. and Vere S. Thompson, Annual Meeting of the American Ceramic Society, New York City, May 1967 (Refractories Division).

## APPENDIX E

## CERAMIC MATERIALS RESEARCH PROGRAM REVIEW

November 1-2, 1966

With the consent of our NASA technical monitor, invitations were extended to interested research personnel to attend the autumn program review. Invitations were sent to appropriate individuals in all federal agencies and to officials of Pacific Coast ceramic industry, aerospace industry and non-profit research laboratories thought to have an interest in the program.

The program in the first day consisted of 15-20 minute briefings by the various research supervisors and the resulting discussion. On the second day, the attendees visited the various faculty members in their laboratories and discussed their research in detail.

Verbal and written comments by attendees indicated the program was mutually successful and most indicated that consideration should be given to periodic repetition of this type liaison. Several indicated that they were able to obtain information on research of interest one to three years earlier than might be possible through normal presentation or publication methods.

## SUMMARY OF ATTENDEES

Visitors	21	(11 on second day)
Faculty Supervisors	13	
University Administrators	4	

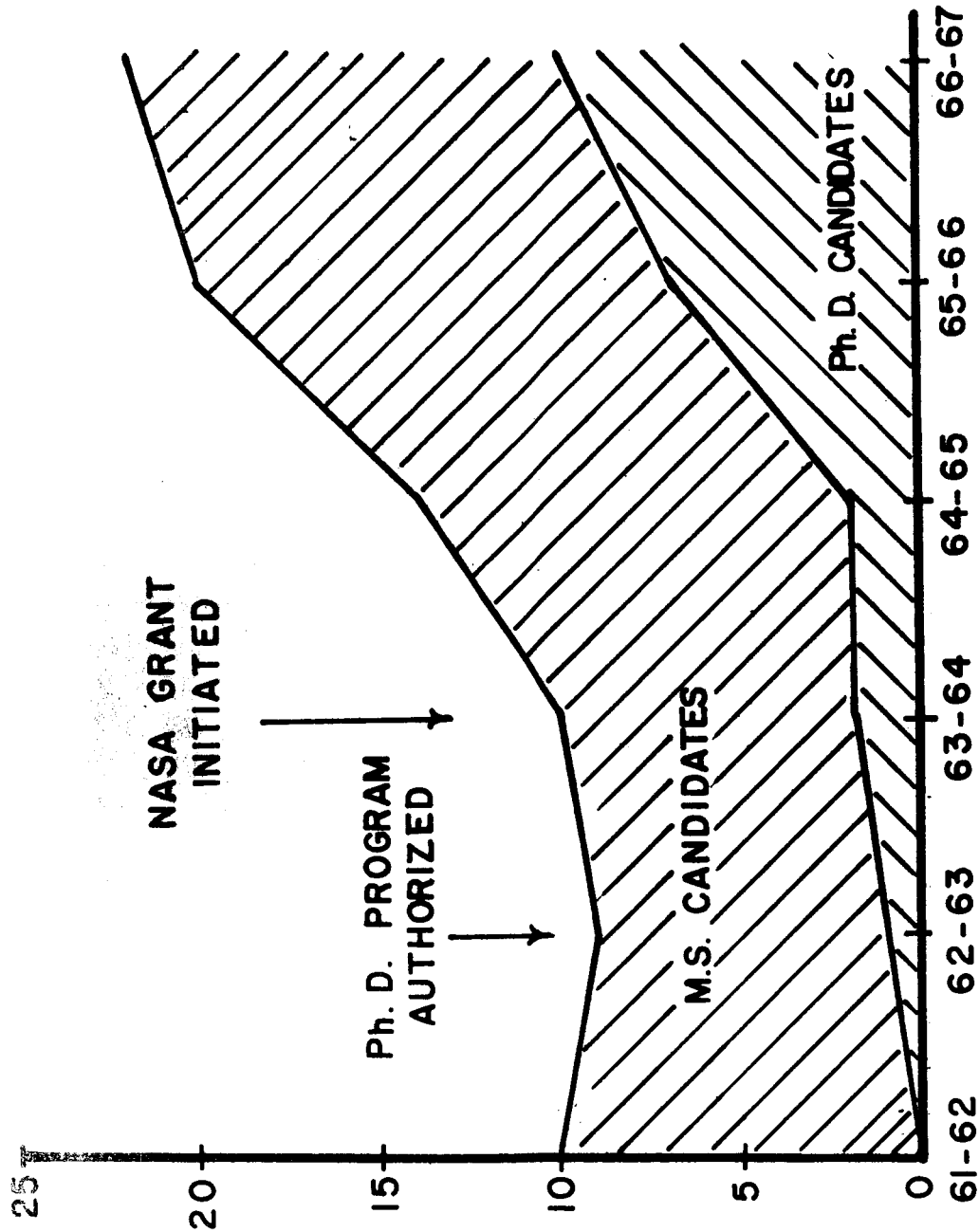
## GEOGRAPHICAL SUMMARY

Washington	8
Oregon	5
California	3
New York	2
Washington, D. C.	2
Ohio	<u>1</u>
TOTAL	21

## EMPLOYMENT SUMMARY

Ceramic Industry	7
Aerospace Industry	5
Federal Agencies	4
USAF (2)	
NASA (1)	
USBM (1)	
Non-Profit Research	3
Educational Institutions	<u>2</u>
TOTAL	21

## APPENDIX F



EFFECT OF NASA GRANT NSG - 484 UPON  
GRADUATE ENROLLMENT IN CERAMIC ENGINEERING,  
UNIVERSITY OF WASHINGTON

## APPENDIX G

## Distribution

Dr. T. K. L. Smull, Director  
Grants and Research Contracts  
Office of Space Sciences, Code SC  
NASA  
Washington 25, D. C.

Mr. James J. Gangler  
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Technology  
NASA  
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Lt. Col. Louis Klinker  
Army Research Office  
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Department of the Army  
Washington 25, D. C.

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Metallurgy Branch  
Army Research Office  
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for Materials  
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Dr. Dan Dedmore  
Lewis Flight Center  
NASA  
Cleveland, Ohio

Dr. George Kendall  
Aerospace Corporation  
El Segundo, California

Mr. D. R. deHalas  
Manager, Materials Department  
Battelle-Northwest  
Richland, Washington

Dr. Henry Heystek  
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